

Certificate of Analysis

Standard Reference Material® 1918

Mercury Porosimeter Intrusion Standard

This Standard Reference Material (SRM) is intended for use in the calibration of mercury porosimeter intrusion analytical instruments. A unit of SRM 1918 consists of one vial containing approximately 12 g of an extruded silica-alumina compound.

Certified Value: The results are expressed as the certified values \pm the expanded uncertainties (Table 1). The certified values are weighted averages [1] of measurements based on intrusion curves from six laboratories. The expanded uncertainties, at the 95 % level of confidence are calculated as $U = ku_c$, where u_c is a combined standard uncertainty for the average, calculated according to the ISO Guide [1,2], and k = 2 is the coverage factor. The value of u_c in each case includes both a combined estimate of the variation of the averages attributed to each source and an allowance for differences among those averages.

Table 1. Certified Values for SRM 1918

Mean Pore Diameter $8.847 \pm 0.363 \text{ nm}$ Median Pore Diameter $8.503 \pm 0.218 \text{ nm}$ Total Intruded Volume $0.547 \pm 0.018 \text{ mm}^3/\text{g}$

Expiration of Certification: The certification of this SRM is considered valid until **01 October 2010**, within the uncertainty specified, provided that the SRM is handled and used in accordance with the Instructions for Use section of this certificate. The certification is considered nullified if the SRM is damaged, contaminated, or otherwise modified.

Stability: This material is considered to be stable during the period of certification. NIST will monitor this material and will report any significant changes in certification to the purchaser. Return of the attached registration card will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was provided by S. Jahanmir of the NIST Ceramics Division.

Analytical measurements for the certification of this SRM was performed by D.B. Minor of the NIST Ceramics Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Support aspects in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Group by B.S. MacDonald.

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Gaithersburg, MD 20899 Certificate Issue Date: 08 July 2002

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INSTRUCTIONS FOR USE

The following is the suggested procedure for preparation of this SRM prior to analysis; pre-dry the sample in a vacuum oven at 100 °C to 110 °C under a vacuum of at least 100 mTorr for from between 12 hours to 18 hours. Cool the sample to ambient temperature while under vacuum. Back fill the oven with dry nitrogen or argon and seal the sample immediately. Transfer the sample to a 60 °C oven or dissector and leave until analysis. Place approximately 0.25 g of sample in a pre-weighed sample holder and heat to 100 °C for 30 minutes while under a vacuum or at least 50 mTorr. Individual pieces of the sample may be broken to fit the sample holder. After the heating source is removed, allow the sample to cool for 15 minutes to ambient temperature. Backfill the sample holder with dry nitrogen or argon and re-weigh the assembly to determine the "dry" sample weight by difference. Re-evacuate the sample for a period of time necessary to achieve a vacuum of 50 mTorr and back-fill with mercury to ambient pressure over a period of 10 minutes. Perform the analysis in accordance with the instrument manufacturer instructions or the procedures of your laboratory.

Participating Laboratories in the Round Robin

Quantachrome Corporation; Boynton Beach, FL

Chevron Petroleum Technology Company; San Ramon, CA

Oil-Dri Corporation of America, Research & Development; Vernon Hills, IL

Grace Davison Chemical, Analytical Services Center; Baltimore, MD

Engelhard Corporation; Beachwood, OH

National Institute of Standards and Technology, Ceramics Division; Gaithersburg, MD

REFERENCES

- [1] Taylor, B.N.; Kuyatt, C.E.; Guideline for Evaluating and Expressing the Uncertainty of NIST Measurement Results, NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC, (1994); available at http://physics.nist.gov/Pubs/. See also Heckert, A.; Filliben, J.J.; Dataplot Reference Manual, http://www.itl.nist.gov/div898/software/dataplot/
- [2] Rukhin, A.L.; Vangel, M.G.; *Estimates of a Common Mean and Weighted Means Statistics*, Journal of American Statistical Association, Vol. 93, No. 441, pp. 303-308 (March 1998).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

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